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L3: Entry 3 of 12

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TITLE: Composition membrane for separating water from fluids containing organic components by means of pervaporation

Brief Summary Text (26):

After applying the polyvinyl alcohol solution to the porous backing layer, crosslinking (pre-cross-linking) takes place during drying under the action of the crosslinking agent. Elevated temperatures between room temperature and 200.degree. C. accelerate the drying and cross-linking. The temperature will normally lie in the range of 80.degree. C. to 180.degree. C., preferably 100.degree. C. to 150.degree. C. The cross-linking time is in the range of 1 to 60 min., preferably 5 to 30 min. The addition of mineral acids, for example sulfuric acid or hydrochloric acid accelerate the cross-linking action. The acids can be added directly to the polyvinyl alcohol solution, which already contains the cross-linking agent. The cross-linking takes place through etherification, esterification or acetalization of the hydroxyl groups of the polyvinyl alcohol where these reactions can also take place simultaneously. Suitable cross-linking agents are known to those skilled in the art. A few examples are described in the European Application EP-A 0 096 339. It has proven advantageous in some cases to apply a second solution of polyvinyl alcohol containing mineral acids in a second step. Here, the solution containing the cross-linking agent is first applied to the porous backing layer and dried at a temperature of 20.degree. C. to 100.degree. C., preferably 50.degree. C. to 80.degree. C. for 2 to 10 min., preferably 4 to 5 min. A second layer is applied to the pre-dried film of polyvinyl alcohol, which consists of a polyvinyl alcohol solution of 2 to 5 wt. %, preferably about 3.5 wt. % and a mineral acid, preferably sulfuric acid and/or hydrochloric acid in a mole ratio of 0.0001:1 to 0.1:1 to preferably about 0.01:1 of acid to polyvinyl alcohol monomer.

Brief Summary Text (44):

When dehydrating amine solutions, no special measures are necessary with respect to the separating layer of the membranes since the separating layer material, i.e. polyvinyl alcohol, is already resistant against hot amine solutions due to crosslinking.

Detailed Description Text (2):

A polyacrylonitrile solution in dimethylformamide is applied to a polyester fleece of about 100. $\mu$  thickness as reinforcing layer and precipitated in water. The obtained porous membrane has a thickness of about 200. $\mu$  (including the reinforcing layer) an asymmetric pore structure and a retention capability for dextran molecules with a molecular weight of over 20,000. This PAN membrane was dried and covered with a 5% solution of polyvinyl alcohol, which contained 0.05 mole maleic acid per mole of polyvinyl alcohol monomer. After drying at 130.degree. C., the obtained membrane was placed in a refined steel vessel having a volume of 2.5L. 0.1 g of solid 1,2-dibromoethane were located at the bottom of the refined steel vessel. To avoid a premature vaporization of the dibromoethane, the container was cooled to 0.degree. C. and the air was suctioned off down to a residual pressure of 20 mbar. The refined steel vessel was then heated to 150.degree. C. and held at this temperature for 5 hours. After cooling and venting, the membrane was removed and tested in a pervaporation experiment with an acetic acid/water mixture. The water content of the feed mixture was varied between 98% and 10%. The permeate for all feed concentrations consisted of more than 99.8% water, the membrane proved to be stable at temperatures up to 100.degree. C. The permeate flow at 80.degree. C. was

2.6kg/m.<sup>2</sup> .multidot.h at a water concentration of 60% in the feed.

Detailed Description Text (8):

An ultrafiltration membrane of polyacrylonitrile with asymmetric pore structure (porous backing layer) on a polyester fleece (reinforcing layer) was coated with a solution of 6% polyvinyl alcohol in water which contained 0.05 mole maleic acid and 0.5 mole formaldehyde per 1 mole of polyvinyl alcohol monomer. After drying at 140.degree. C., the membrane was placed in a refined steel vessel, it was closed and pumped down to a residual pressure of 20 mbar. Nitrogen was then introduced which was passed through concentrated aqueous hydrochloric acid and thus saturated with hydrochloric, until the pressure in the refined steel vessel was 500 mbar. The container was then heated over 4 hours at 180.degree. C. After cooling and venting, the membrane was used in a pervaporation test with a feed mixture of 98 wt. % acetic butylester and 2 wt. % water. Only water was found in the permeate up to a concentration of 99.9 wt. % acetic butylester. At a feed concentration of 99% ester and a temperature of 90.degree. C., the permeate flow at 145 g/m.<sup>2</sup> .multidot.h was distinctly higher than the flow under the same conditions (100 g/m.<sup>2</sup> .multidot.h) for the comparative membrane described in Example II, by which 35 wt. % acetic butylester was found in the permeate.

Current US Cross Reference Classification (1):

210/500.42

CLAIMS:

8. Composite membrane of claim 1, characterized in that the polyvinyl alcohol layer subjected to post-cross-linking has been obtained by pre-crosslinking by means of etherification, esterification and/or acetalization.